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INVESTIGATION OF FISH-SCALE PHENOMENA*

By W. W. Higgins and W. A. Deringer

Abstract

Some of the characteristics of fish scaling are discussed, and the writers establish the fact that fish scaling is always accompanied by the evolution of gas. Three factors, which were believed to influence the tendency to fish scale and which were investigated, are (*1*) *the base metal:* the metallurgy of the steel and the effect of cold-working on steel; (*2*) *the clay in the enamel:* the effects of various types of clays on fish scaling; and (*3*) *the furnace atmosphere:* the effects of various furnace atmospheres and of water vapor in the furnace atmosphere.

I. Introduction

At the beginning of this investigation several years ago, fish scaling, which showed the peculiarity of occurring along the edges of the steel sheet, was encountered in production work. The steel sheet used was the hot-rolled, finished "rimmed" type, from 1/4 to 3/4 in. thick. Its range of chemical composition was carbon under 0.20, manganese under 0.50, phosphorus under 0.02, and sulfur under 0.03%.

Shop records, which were carefully made on the occurrence and location of fish scaling for hundreds of tons of this steel, bore out the original finding, that is, when fish scaling occurred on this material, it was most likely to appear along the edge of the steel sheet.

A production study of results on the commonly used low-metalloid content enameling iron (also of the rimmed type), 0.090 to 0.250 in. thick, showed the same tendency.

The problem then confronted the laboratory to find some way of changing the enameling process to eliminate this tendency to fish scale. Although all of the variables named were not under suspicion at the start of the investigation, the results of initial laboratory trials indicated that the factors which should be investigated were (1) the base metal, (2) the clay in the enamel, and (3) the furnace atmosphere.

This report, therefore, is divided into two sections, the first of which shows the characteristics of the fish-scaling phenomena and the second, the factors which have some bearing on the fish-scaling tendency.

II. Characteristics of Fish-Scaling Phenomena

(1) The Base Metal

Some of the characteristics of fish-scaling phenomena which the authors have observed during their studies are (*a*) fish scaling is always accompanied by the evolution of some gas or gases; (*b*) the tendency to fish scale is much worse when both sides of the piece are enameled; and (*c*) there is only one type of fish scale.

In connection with characteristic (*a*), an enameled plate which had not yet fish scaled was placed in a shallow pan. Enough mineral oil was added to the pan to cover the enameled surface of the plate to a depth of 1/8 in. As soon as a fish scale presented itself, a large gas bubble, visible to the naked eye, appeared and rose to the surface of the oil, and a train of tiny bubbles could be seen under the microscope to travel from the bottom of the fish scale to the surface of the oil. These bubbles continued to erupt from the fish scale for some time.

On one particular fish scale, the bubbles were still being given off after four hours of continuous gas evolution. It was decided to carry this phase of the investigation further and to attempt to get a sample of this gas and analyze it. A few trials helped to develop a satisfactory technique, and the following procedure was adopted: Fifty plates of 0.105-in. thick steel, 3 by 6 in., which was known to have a fish-scaling tendency, were enameled on both sides and sprayed with enamel containing 6% of Florida kaolin, a "clear" clay which tends to promote fish scaling. All of these plates were fired simultaneously for 12 minutes at 1620°F. As soon as they were cool enough to handle, the plates were stacked in a desiccator which had a stopcock in the lid. The ground glass joint was sealed with sealing wax all of the way around. Sealing wax was also used to seal the glass tube leading from the top of the desiccator to a gas-extraction apparatus. This apparatus (Fig. 1), which consisted of two mercury vapor pumps, three oil pumps, and a McLeod gauge, was capable of creating a vacuum of less than 0.001 mm. of mercury.

The desiccator was evacuated until the McLeod gauge showed a pressure of 0.001 mm., the stopcock in the desiccator lid was turned off, and the pumps were shut off. After allowing the evacuated desiccator to stand for 24 hours, the McLeod gauge showed considerable increase in pressure in the desiccator. This gas was drawn off, collected over mercury, and analyzed with a precision Burrell gas analyzer which is capable of analyzing samples of gas as small as 0.5 cc. (Fig. 2).

The sample of gas (which amounted to 24.6 cc.) was analyzed for oxygen, carbon dioxide, hydrogen, carbon monoxide, illuminants, and unsaturated hydrocarbons; the remainder was assumed to be nitrogen. The analysis of two gas samples gave the following results:

* Presented at the Forty-Third Annual Meeting, The American Ceramic Society, Baltimore, Md., April 2, 1941; received May 22, 1941; revised copy received September 17, 1941.

Gas	Sample No. (1)	(2)
Carbon dioxide	0.0	0.0
Oxygen	0	0
Carbon monoxide	0	0
Hydrogen	97.7	98.6
Unsaturated hydrocarbons	0	0
Illuminants	0	0
Nitrogen	2.3	1.4

These analyses agree closely with the findings of Hoff and Klärding,[1] whose analysis of 1 cc. of gas extracted from enameled plates showed this gas to be 85 to 90% of hydrogen. The analyses in the present studies were for gas samples considerably larger than that checked by Hoff and Klärding[1] so that a greater degree of accuracy was possible.

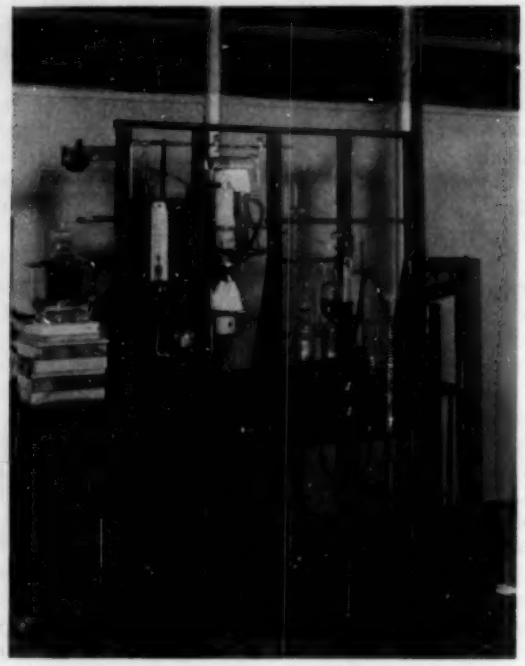

FIG. 1.—Gas-extraction and collection equipment.

The volume of gas collected, which was calculated on a percentage basis, was 1.42% of the volume of the steel. If this gas was considered to be atomic hydrogen, the volume percentage was 2.84% although it was only 0.000018% by weight. The amount of gas given off through the fish scales and collected was known to be but a part of the total gas present in the steel, inasmuch as the test specimens started to fish scale before it was possible to evacuate and seal off the desiccator for the collection of the gas sample.

The tendency to fish scale was greatly reduced if the enamel was applied only to one side of the piece or if a dust coat was applied to the back of the piece. McIntyre remarks about the freedom from defects on an enameled surface when no enamel is applied to the back side.[2] This fact concurs with the theory that fish scaling is the result of gas pressure at the metal-enamel interface.

Observations in the laboratory led the authors to believe that there is only one kind of fish scale. Both

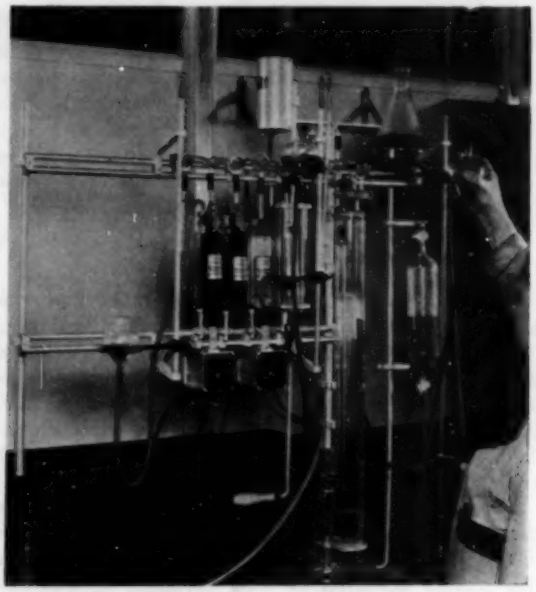

FIG. 2.—Precision Burrell gas-analysis apparatus.

the size of the fish scale and the time required for its occurrence increased with the thickness of the coating. The so-called "shiner" scale appeared on thin coatings and immediately after firing, whereas the "delayed" fish scales occurred only on the heavier coatings and several hours after firing (Fig. 3).

The same results were produced artifically. The plates were enameled with various thicknesses of enamel, ranging from very thin to very heavy coats. No enamel was applied to the back sides of these samples. The specimens were pickled on the back with a 5% solution of hot sulfuric acid until fish scales appeared on the other side. The piece with the lightest coat of enamel was the first to fish scale, and the defects were small and of the shiner-scale variety. Specimens with intermediate thicknesses of coating soon displayed intermediate-sized fish scales, and still later, the specimens with heavy coatings revealed large fish scales. So in two different ways, the same results were obtained. There was only one kind of fish scale, but it varied in size (and in the time required for it to put in its appearance) with the thickness of the enamel coating (Fig. 4).

III. Factors Studied

As outlined in section I, the three factors studied in this investigation were (1) the base metal, (2) the clay in the enamel, and (3) the furnace atmosphere.

[1] H. Hoff and J. Klärding, "Fish Scaling Related to Gas Evolution in Enameling," *Stahl u. Eisen*, **58**, 914–16 (1938); *Ceram. Abs.*, **18** [5] 121 (1939).

[2] C. A. Zapffe and C. E. Sims, "Relation of Defects in Enamel Coatings to Hydrogen in Steel," *Jour. Amer. Ceram. Soc.*, **23** [7] 187–221 (1940); G. H. McIntyre discussion, [10] p. 298.

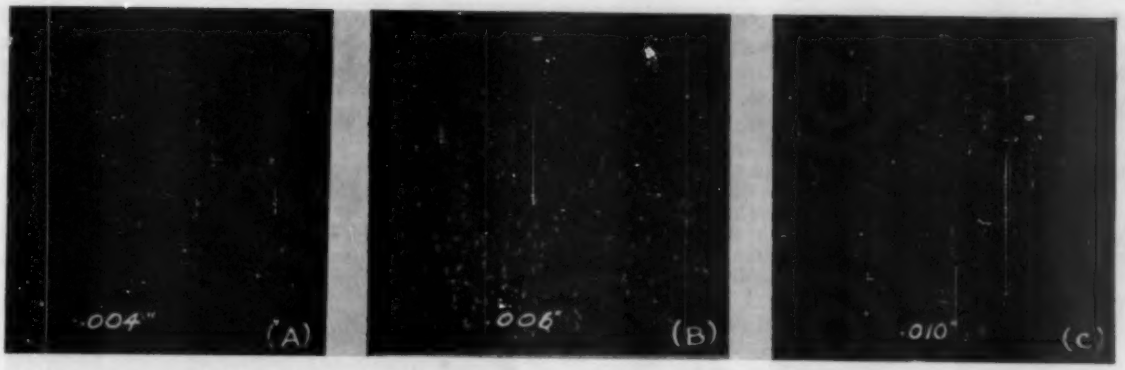

FIG. 3.—Enameled plates of various thicknesses of coating showing relation between thickness of coating and size of fish scales.

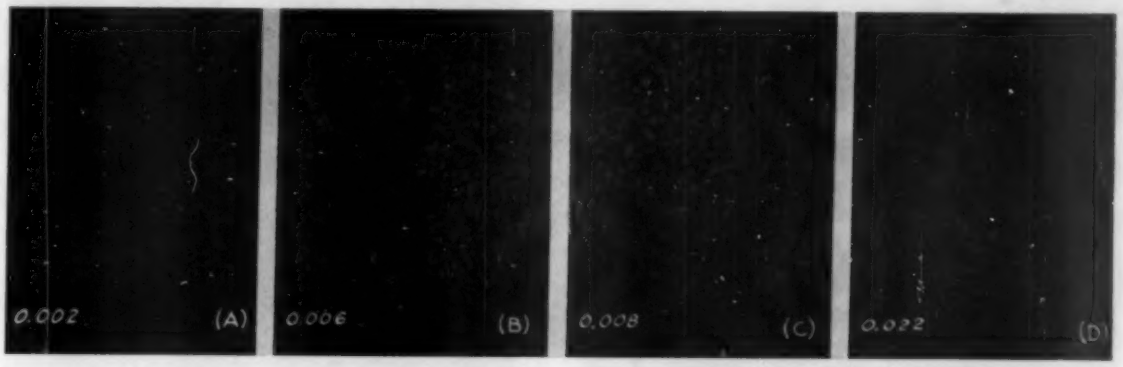

FIG. 4.—Fish scales produced by pickling back side of plates enameled with coatings of various thicknesses.

(1) The Base Metal

Sheets used by the porcelain enameling industry are ordinarily made of very low carbon, open-hearth steel (under 0.05% of carbon), finished, and cast into ingots by the "rimming" process.[3] The surface of these ingots (which cools and solidifies first) has a lower carbon and impurity content than the interior. This sound surface layer may be several inches thick on a large ingot and is responsible for the relative freedom from seams, scabs, and other defects on the surfaces of the finished sheets. The interior of the ingot, which is called the core, is characterized by internal blowholes, which constitute from 2 to 4% of the total volume and are the result of the evolution of carbon monoxide gas during the solidification of the ingot.

Figure 5 is a deep-etched section through an ingot, showing the rim and core areas. When the ingot is cut in slabs and rolled into sheets, the core remains sandwiched between two layers of rim steel. The core does not extend entirely to the edge of the sheet on all four sides. Along one or more edges (depending on the fabrication), the sheet is entirely solid rim for a distance of from 2 to 10 or more inches in from the edge, depending on the size of the sheet and on the amount of side scrap removed by the steel mill.

It had been known for some time at this laboratory that the enameled "solid-rim" portion of a steel sheet had much more tendency to fish scale than the "rim-and-core" portion; therefore, when this problem presented itself, the first step was to determine whether or not the defect was present only in the solid-rim portion of the sheet. This was done by taking sections through fish-scaled portions and also through those parts free from fish scale. These sections were then deep-etched by pickling for 20 minutes in a 50% solution of boiling hydrochloric acid. The resultant etch showed that, although the steel was predominantly rim and core, fish scales occurred only on the solid-rim portion of the sheet (Fig. 6), which verified previous tests made at this laboratory. These findings, however, did not show that all solid-rim steel would fish scale; such steel from another source did not fish scale under the same conditions, but regardless of the source of the enameling stock, the solid-rim part of the sheet always had a greater tendency to fish scale than the rim-and-core portion.

No fish scales were present on seamless tubing made of low carbon "killed" type steel, which had been cold-reduced, whereas the same steel fish scaled severely when it had not undergone cold reduction (Fig. 7). Plates that were sandblasted severely, moreover, had less tendency to fish scale than those that were given a light sandblast. Both of these observations seem to

[3] R. H. Heyer, Engineering Physical Metallurgy, p. 178. D. Van Nostrand Co., Inc., New York, N. Y., May, 1939.

indicate that a certain amount of cold reduction is beneficial, and it also focuses attention on the fact that the amount and severity of the sandblasting operation is a variable which must be controlled. These findings agree with the work of others. Zapffe and Sims[4] say,

> Evidence from several different sources indicates that the structure of metals is a mosaic or block structure, and density measurements have shown that the specific volume of metal is increased by cold-working. This increase in the volume can only be due to an increase in the volume of the submicroscopic pore spaces, which action would provide greater internal free space in which hydrogen might collect.

In a recent publication, Zapffe and Faust[5] say,

> Differences have been noted in the enamel industry where cold-worked material generally shows less tendency toward developing hydrogen-caused defects than annealed metal, in contrast to the experience in electroplating. The effect of cold-working, simply stated, is that the deformation opens the structure of metals and thereby accelerates both absorption and effusion of hydrogen, depending on the direction of the concentration gradient of hydrogen atoms within, and in contact with, the iron lattice. The reason that enamelers find cold-worked metal preferable may be explained by noting that enameled ware is dried or baked or slowly heated before firing so that hydrogen removal would be maximum for cold-worked metal.

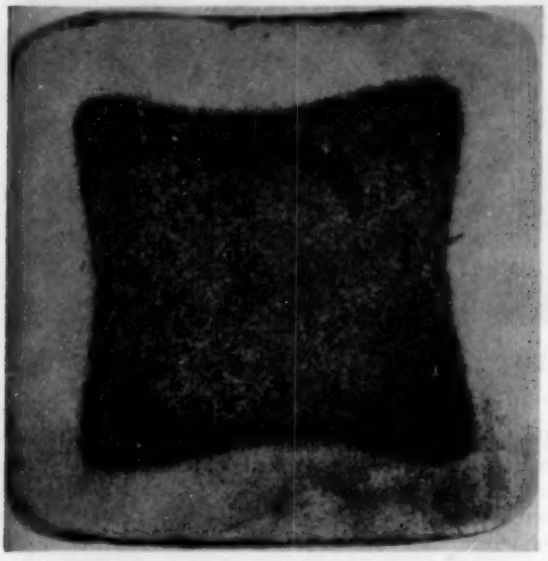

FIG. 5.—Section through ingot of rimming steel showing rim and core.

The enamel coating on a hard-sandblasted surface contained more bubbles than the enamel on the light-sandblasted areas, which may be seen in the photomicrographs (Fig. 8); (*A*) is a section through the enamel over a normal sandblasted area; (*B*) is an etched section through a normally sandblasted surface; and (*C*) and (*D*) are the same sections over a hard-sandblasted area. Perhaps the "effusion of hydrogen"[5] from the cold-worked metal takes place while the enamel is molten and produces the bubble structure, whereas light sandblasting leaves the metal in the "as-rolled" condition, and the evolution of the gas is thus retarded until later when the enamel has become rigid. Figure 9 shows a plate that was submitted to severe sandblasting on one small area, which is the only part of the plate that did not fish scale.

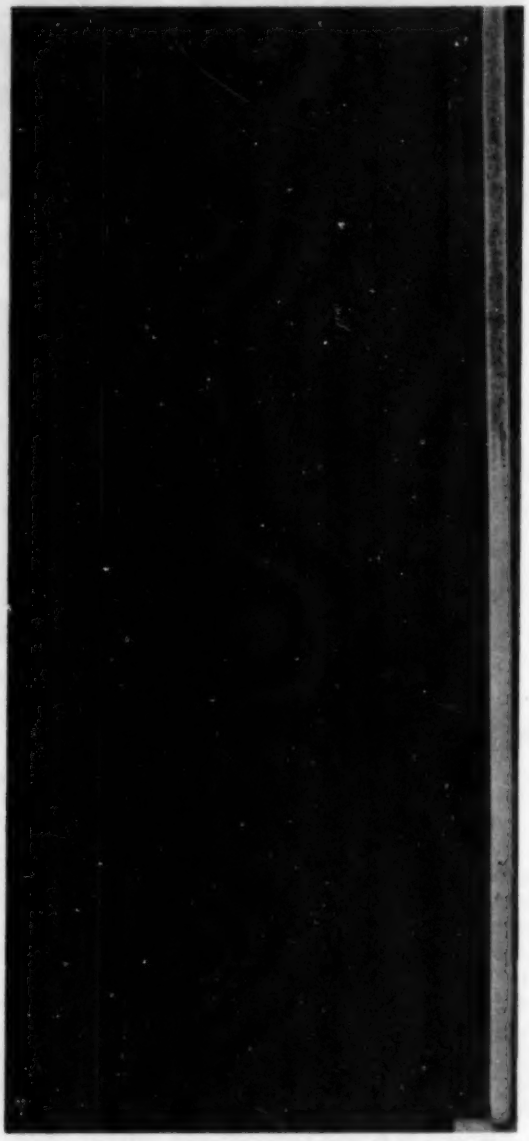

FIG. 6.—Enameled plate showing effect of steel structure on fish scaling; etched section at right shows that fish scaling occurred only in solid-rim portion of plate.

Note: All of the test samples made during this investigation were hot-rolled finish enameling stock and were sandblasted before enameling, which is the method used in this plant to prepare the metal surface. No comparison therefore was made of the effect of pickled surface versus sandblasted surface on the tendency to fish scale.

[4] C. A. Zapffe and C. E. Sims, "Hydrogen, Flakes, and Shatter Cracks," *Metals and Alloys*, **11** [5] 145–51; [6] 177–84 (1940); **12** [7] 44–51; [8] 145–48 (1940).

[5] C. A. Zapffe and C. L. Faust, "Metallurgical Aspects of Hydrogen in Electroplating," *Proc. Educ. Sess. Amer. Electroplaters' Soc.*, **28**, 1–20 (1940).

(2) The Clay in Enamel

It has been known for some time that some clays must not be used in ground coats because of their tendency to promote fish scales. A Florida kaolin was found to have this effect. It had good suspending properties and gave little opacity to the fired enamel, a so-called "clear-firing" clay. For a comparison of fish-scaling tendencies, millings were made in which twenty other commercial enamel clays were substituted for this kaolin. These clays included clear-firing kaolins, blends of domestic clays, Tennessee ball clay, Kentucky ball clay, and an English ball clay.

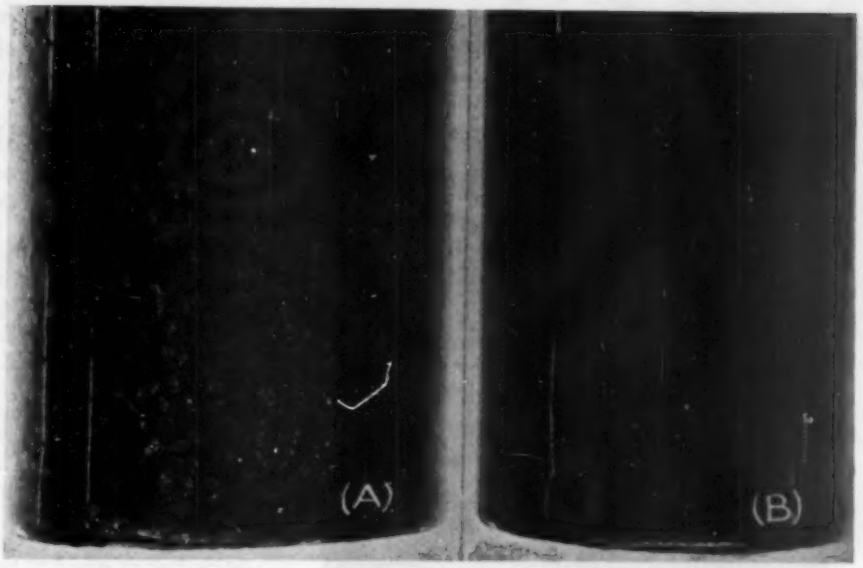

FIG. 7.—(*A*) Hot-drawn and (*B*) cold-reduced seamless tubing made from "killed" type steel.

Plates of solid-rim enameling stock, which was known to have a tendency to fish scale, were sprayed with enamel from these various millings. The amount of sandblasting, the spray weight, and the firing conditions were maintained constant, and the plates were fired, cooled, and annealed at 400°F. for 30 minutes to accelerate the delayed fish scaling. The specimens were then compared for fish scaling and were examined under a microscope to compare the enamel bubble structures.

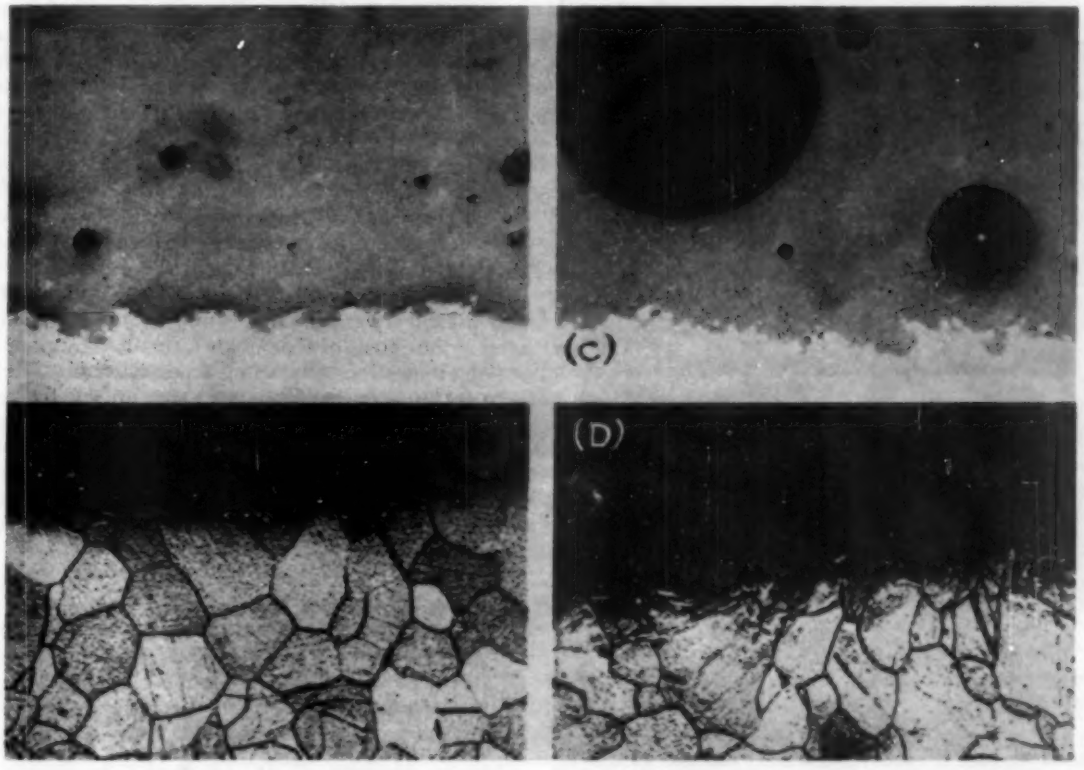

FIG. 8.—Photomicrographs showing effect of (*A*) ordinary and (*B*) severe sandblasting on enamel bubble structure; ×300.

This experiment demonstrated the importance of the type of clay used. The plates ranged from no fish scales at all to a solid mass of fish scales. The kaolins, in general, showed the most fish scales; and the Tennessee ball clays and English ball clay, the least of the unblended clays. Plates that were coated with enamel containing German Vallendar, one of the Tennessee ball clays, or one of the domestic blended clays were entirely free from fish scales.

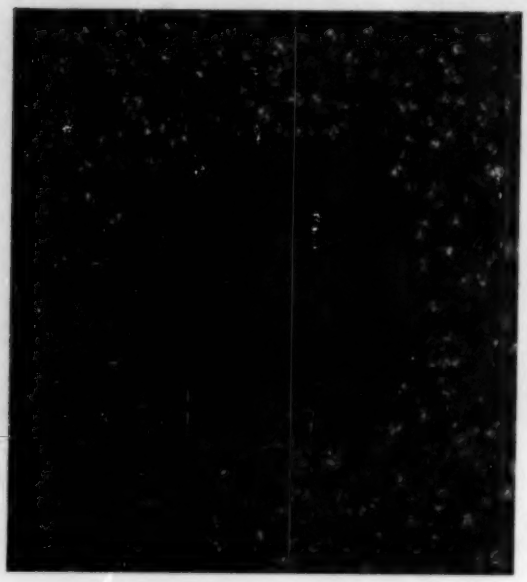

FIG. 9.—Effect of severe sandblasting (spot in center) on fish-scaling tendency.

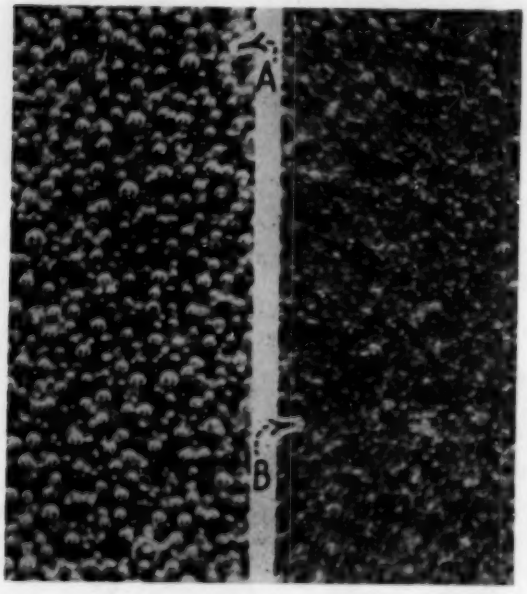

FIG. 10.—Photographs (*A*) of enamel containing opaque-firing or bubble-producing clay; (*B*) clearing-firing clay; ×50.

To compare the bubble structures imparted to the enamels by the different clays, a microscopic examination revealed that although all of the enamels had some bubble structure the enamels which showed the least fish scaling were those containing the most bubbles (Fig. 10).

Hoff and Klärding[1] emphasize the importance of the chemically combined water of the clay and explain that this water breaks down at high temperatures in the presence of iron, according to the equation,*

$$3Fe + 4H_2O \longrightarrow Fe_3O_4 + 8H.$$

On the basis of this equation, it was decided to determine whether or not the various clays lost their water at different rates during the heating period in the furnace. Dehydration curves were made of these clays by heating samples of the clays for 1 hour at a time at 100° intervals from 300° to 1400°F. The weight loss on heating was assumed to be all water loss because all of these clays were relatively pure and low in organic matter. After the first trial, the interval between 700° and 1400°F. was the only portion of the dehydration curves to be considered; below 700°F., the water loss was found to be small and approximately the same for all clays. After it was ascertained that most of the water loss took place at or below 1000°F., the intervals were changed to 50° between 700° and 1000°. These curves (Fig. 11) show a striking relationship between the rate of water loss and the fish-scaling tendency. Those clays which were associated with bad fish scaling lost the most water between 900° and 1000°; those which lost the least water during this interval had the least tendency to cause fish scaling. A definite relationship seemed to exist between fish scaling and the amount of water loss of the clay during the firing of the enamel.

Another relation also exists, which offers a more logical explanation of the tendency to fish scale, namely, that between bubble structure in the enamel and the fish-scaling tendency. As shown in Fig. 10, the type of clay used in the enamel is the governing factor in determining the extent of the bubble structure. An investigation of numerous clays of all types shows this definite relationship between the bubble structure and fish-scaling tendency. Those clays which produced a good bubble structure in the fired enamel coating gave the enamel good resistance to fish scaling in every case; conversely, the use of clear-firing clays resulted in enamels with pronounced fish-scaling tendencies. Harman[6] says,

> The mineralogical composition of clays has a direct bearing on the water loss when heated. This is a property which has received but little attention by enamelers. It would appear that this property of clays would be of great importance in connection with the firing of enamels and would possibly have some bearing on the appearance of the fired enamel.

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* See p. 197 of footnote reference 2.

[6] C. G. Harman, "Clay Technology as Applied to Enamels," *Proc. Porcelain Enamel Inst. Forum, Oct., 1940*, pp. 20–25; *Ceram. Abs.*, **20** [4] 84 (1941).

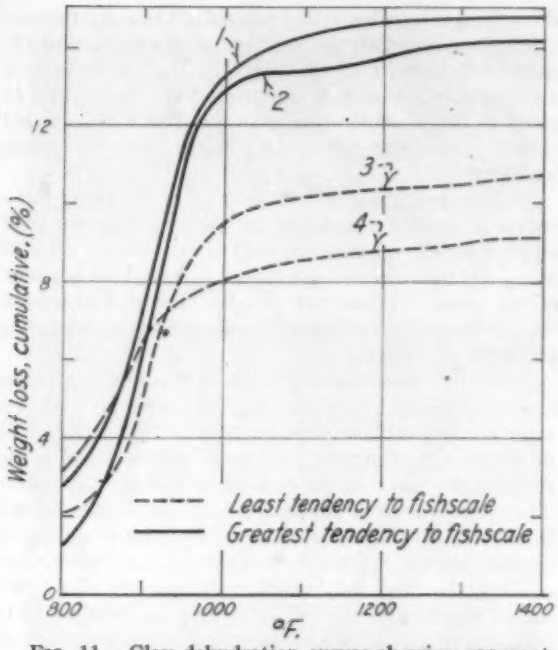

FIG. 11.—Clay dehydration curves showing apparent relation between weight loss and fish-scaling tendencies for various clays; curve (1) Florida kaolin; (2) Georgia kaolin; (3) Tennessee ball clay; (4) German Vallendar clay.

It was decided to follow up Harman's suggestion and attempt to correlate the mineralogical content of the various clays with the fish-scaling tendency. Some millings were made using 1.25% of bentonite in place of the regular 6% clay addition; in other millings, 3½% of a high illite-bearing clay was substituted. These enamels were applied to solid-rim steel and fired in air in the normal manner. The plates sprayed with enamel containing bentonite and those sprayed with enamel containing the illite clay fish scaled almost as badly as the enamel milled with the kaolin, and it was concluded that apparently no relationship exists between the clay mineral content and the fish-scaling tendency.

As a further check on the effect of the clay minerals, additional plates were enameled with the same enamels and dried for 2 hours at 600°F. This process theoretically should have removed 18% of the 23% chemically held water in the bentonite and about 4% of the 8% chemically held water in the illite clay. The results, however, did not indicate any decrease in fish-scaling tendency. The plates that were dried at 600°F. fish scaled just as readily as those dried at 200°F.

At the suggestion of Andrews,* the role of the organic matter in the clay was investigated. Because different clay minerals absorb different amounts of organic matter as has been stated, it was thought that the different bubble structures imparted to the enamel by the different clays might be due to the organic matter present. A milling, using the kaolin or clear-firing clay, was made with the following mill additions: frit 100, kaolin 6, methylene blue 0.075, sodium nitrate 0.075, ammonium chloride 0.3, and water 45 parts. The sodium nitrate was added to oxidize the organic matter (methylene blue), and the ammonium chloride was added to fine out the larger bubbles.

Plates of solid-rim steel, sprayed with this enamel and fired, showed no fish scales, and a microscopic examination revealed that this mill addition had given the enamel a bubble structure similar to that produced by the enamel containing German Vallendar clay.

Millings were also made, using the methylene blue (without the ammonium chloride and sodium nitrate) and with ammonium chloride and sodium nitrate (without the methylene blue). The fired samples indicated that the presence of the organic matter was necessary to prevent fish scales. Here again, the fish-scaling tendency was reduced by imparting a bubble structure to the enamel.

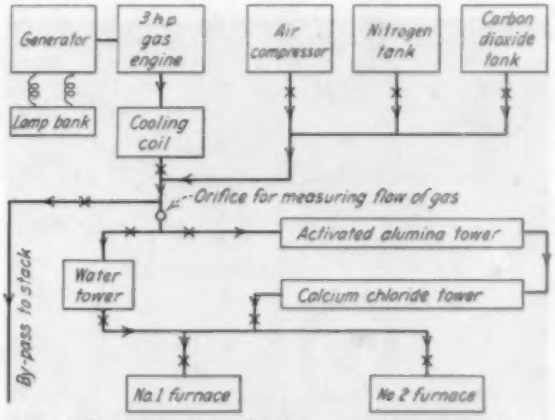

FIG. 12.—Flow sheet showing method used to obtain various furnace atmospheres employed in this investigation.

This synthetic alteration of the properties of a clay opens up a new field of investigation. (Perhaps the time will come when clays can be "made-to-order.")

(3) The Furnace Atmosphere

Past experience in this and in other laboratories has shown the necessity of oxygen in the furnace atmosphere if good bond between the base metal and the enamel is to be obtained. Past research at this laboratory has shown, furthermore, that the actual control of the amount of oxidation of the steel surface during firing eliminates or minimizes certain defects and a patent has been granted covering this procedure.[7] During the course of the experimentation, a plate of solid-rim steel was sprayed and fired in an oxygen-free atmosphere after it had been oxidized sufficiently to give good bond; this plate did not fish scale. It was then decided to investigate the role of the furnace atmosphere in this problem.

* A. I. Andrews, University of Illinois.

[7] (*a*) W. G. Martin, "Method of Enameling," U. S. Pat., 2,004,632, June 11, 1935; *Ceram. Abs.*, **14** [8] 184 (1935); (*b*) W. G. Martin, "Enamel Defects Prevented by Firing in Inert Gas Atmosphere," *Ceram. Ind.*, **25** [1] 20–21 (1935); *Ceram. Abs.*, **14** [9] 210 (1935).

The usual laboratory method of firing in a controlled atmosphere is to oxidize the plates at 1000°F. for the required length of time (depending on the gauge of the metal) and then to transfer the plates immediately to another furnace, where the enamel is fused down at the proper temperature. The second furnace has an oxygen-free atmosphere so that no further oxidation of the metal takes place.

The plates used in this investigation were 6 by 8 by 0.150 in. The proper firing time was found to be 10 minutes of oxidation at 1000°F. and firing for 12 minutes at 1625°F.

In investigating the effects of the furnace atmosphere on the fish-scaling tendency, the pertinent questions were (1) does the amount of water vapor in the furnace atmosphere have any effect on fish scaling and (2) will an oxygen-free atmosphere in the firing chamber produce the same results as firing in air?

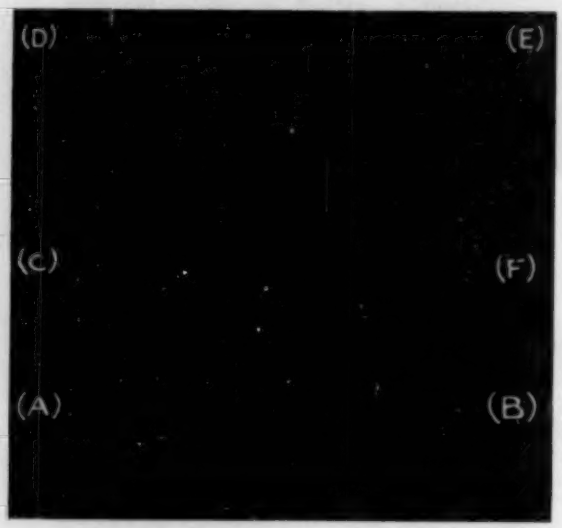

FIG. 13.—Solid rim plate photographed after enameling for second time; *AB*, firing bar mark of previous coat; *CDEF*, area brushed free of enamel on previous coat.

The following program was outlined to determine the answers to these questions. Solid-rim plates, 6 by 6 by 0.150 in. in size, were sprayed with enamel containing Florida kaolin to a thickness of 36 gm. (wet) per sq. ft. One of these plates was fired under each of the following conditions: (*a*) *Fired in air:* (i) water vapor of furnace atmosphere, low; dew point of 15°F. (1/2% of water vapor) and (ii) water vapor of furnace atmosphere, high; dew point of 130°F. (15% of water vapor); and (*b*) *fired with oxidation control:* (i) water vapor of furnace atmosphere, low; dew point of 15°F. (1/2% of water vapor) and (ii) water vapor of furnace atmosphere, high; dew point of 130°F. (15% of water vapor). Figure 12 is a flow sheet showing how the various furnace atmospheres were obtained.

The results of the tests conducted under (*a*) and (*b*) are as follows: (1) the plates fired by oxidation control showed no fish scales, either when fired in a dry furnace atmosphere or when the dew point was high, and (2) the plates fired in air containing a low percentage of water vapor fish scaled much less than those fired in air with high water content; the latter fish scaled violently, and the fish scaling began while the plates were still quite hot.

An unusual phenomenon was observed during the course of this investigation. A plate of solid-rim steel was enameled, blasted off, and re-enameled; after it was cool, two rows of fish scales appeared on one side of the piece. These two streaks extended crosswise on the specimen and were the same distance apart as the firing bar marks.

To determine whether or not these fish scales had been caused by firing bar marks, another piece of solid-rim steel was enameled and fired. Fish scales appeared all over the enamel surface, and notches were filled in the edges of the piece at the ends of each firing bar mark. The piece was then sandblasted off, re-enameled, and fired so that the firing bar marks of this test would occur on the reverse side from those of the previous test. After cooling, the piece fish scaled more severely along two lines extending between the notches. Further tests revealed that if a piece were enameled and blasted off several times more severe fish scaling would occur only over the firing bar marks of the last previous coat.

It was suspected at this point that the amount of oxidation of the base metal had been instrumental in causing these streaks of fish scales. This reasoning was substantiated by the fact that the control of oxidation in the firing operation had been found to inhibit fish scaling. The next step consisted in enameling a piece of solid-rim steel and brushing one half of the back side free of enamel; after it was fired, the piece was sandblasted off and re-enameled. On this second coat, however, none of the enamel was brushed off the back side. After it was fired and cooled, fish scales appeared, but they were much more severe over the previous firing bar mark and on the area which had been brushed free of enamel on the previous coat (Fig. 13). The increased amount of oxidation which had taken place on the enamel-free area during the firing of the first coat obviously had not been entirely removed by the subsequent sandblasting operation, and this oxide had contributed in some way to the formation of fish scales. A microscopic examination revealed that the enamel coating contained many more bubbles in the areas which had few fish scales than in those areas that showed severe fish scaling.

The following test was carried out to investigate additional effects of iron oxide on fish scaling. Seven plates of solid-rim steel were sprayed wet to a weight of 40 gm. per sq. ft. with an enamel containing clear-firing clay and were fired in an oxygen-free atmosphere for 10 minutes at 1600°F. after oxidizing at temperatures shown in Table I.

Figure 14 shows photomicrographs taken from specimens No. 1 (*A*), No. 4 (*B*), and No. 7 (*C*). It is apparent from these pictures and from the results given in Table I that an increase in the total oxidation increases the tendency to fish scale and decreases the

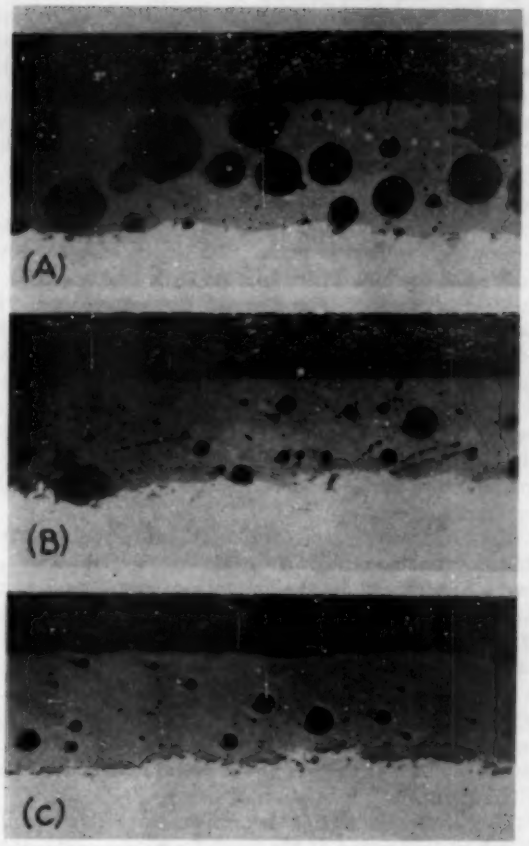

FIG. 14.—Photomicrographs of section through enameled plates subjected to various amounts of oxidation before firing in inert atmosphere; ×100; (*A*) no oxidation; (*B*) oxidized 10 minutes at 1000°F.; (*C*) oxidized for 20 minutes at 1100°F.

number and size of bubbles present in the glass coating.

To check the role of iron oxide further, varying amounts of ferrous iron oxide (FeO) up to 10% were added as mill additions to enamel that contained clear-firing clay. Plates sprayed with these enamels were fired in an oxygen-free atmosphere (see Table II).

All of this experimental evidence points to the importance of the amount of iron oxide present and its role in influencing the tendency to fish scale. Increased amounts of iron oxide apparently bring about a decrease in the bubble structure of the ground coat. Andrews has suggested that this no doubt is due to the fluxing action of the iron oxide, which softens the enamel and makes it more fluid and thus gives an opportunity for the glass to be fined free of bubbles by the time the firing cycle is complete. This experimental work emphasizes the importance of controlling the amount of oxidation and explains why the "oxidation-control" process is so helpful in eliminating fish scales.

TABLE I

Sample No.	Time (minutes)	Oxidation temp. (°F.)	Results (degree of fish scaling)
1	None	None	None; enamel full of bubbles
2	2	1000	None; enamel full of bubbles
3	5	"	Two
4	10	"	Scattered
5	20	"	More than No. 4
6	10	1100	Severe
7	20	"	Very severe; no bubbles

TABLE II

Sample No.	FeO (%)	Results	
		Bubbles	Fish scales
1	0	Extensive structure	None
2	1¼	Fewer than No. 1	None
3	2½	Fewer than No. 2	Three
4	3¾	Fewer than No. 3	Slight
5	5	Slight structure	Scattered
6	7½	None	Severe
7	10	None	Severe

With the knowledge that the amount of iron oxide present is of vital importance, it is best to go back and reconsider the phenomenon of fish scaling over the firing bar marks of a blasted-off enamel coat. An examination of these marks shows that the enamel is rich in iron oxide in this area. Further tests were made using various other metals in place of the regular nickel-chromium-iron alloy ordinarily used to support the ware during the firing operation. Pure nickel, Alumel, Chromel, iron, and constantan were used. All of the firing marks produced by these metals were alike, showing high iron oxide concentrations in the firing bar marks and also fish scaling in these areas when the samples were blasted off and re-enameled. Another piece was enameled and fired, using the edges of two ceramic tile for support. An examination of these firing marks showed no signs of excess oxidation, and the subsequent coat (applied after sandblasting) showed no fish scaling in the areas where the ceramic-tile firing marks had been. As further evidence that the excessive iron oxide concentration is responsible for fish scaling over firing bar marks, the following observation was made: In the foregoing experimental work, the location of the firing bar marks could be seen on the steel after the enamel had been removed by sandblasting. Evidently all of the iron oxide or enamel containing high-iron oxide concentrations had not been removed by sandblasting. Plates in this condition invariably fish scaled over these marks when they were re-enameled; if the pieces, however, were given two or three extra "passes" by the sandblaster, the marks could not be seen, and the pieces would not fish scale preferentially over these marks when they were subsequently enameled.

A careful analysis of the foregoing tests shows that in every instance these data substantiate the theory that high-iron oxide concentrations tend to eliminate the bubbles in the glass and for this reason to promote fish scaling.

As a final experiment to prove or disprove the results obtained in the investigation thus far, another test was conducted. A piece of hot drawn, seamless tubing of killed steel (0.05% of carbon), measuring 2½ in. in outside diameter with a wall ⅛ in. thick, was cut

in two equal lengths, and one piece was cold-reduced to $2^1/_4$ in. outside diameter. Each piece was then cut into five 6-in. lengths, enameled, and fired as shown in Table III.

TABLE III

Specimen No.*	Clay	Firing conditions	Results (fish scales)
1H	Domestic blend	Atmosphere control	None
1C	" "	" "	"
2H	" "	Air	Very few
2C	" "	"	None
3H	Fla. kaolin	Atmosphere control	"
3C	" "	" "	"
4H	" "	Air	Bad
4C	" "	"	None
5H	" "	"	Bad
5C	" "	"	None

* H, hot-drawn tubing; C, cold-reduced tubing; one coat was used throughout except for specimens Nos. 5H and 5C, which had two coats.

A careful examination of these results shows that they substantiate the findings already disclosed in this study. Pieces that were fired in a controlled atmosphere or with an enamel containing a blended clay of high fish-scale resistance or on cold-reduced pieces showed less tendency to fish scale than enamel that contained a clear-firing clay applied over hot-rolled steel and fired in air. A combination of two or more of the fish-scale retarding factors further decreased the tendency to fish scale.

IV. Summary

The following conclusions result from the foregoing experiments:

(1) Enamels fish scale more readily over the solid-rim portion of rimmed-type sheet steel.

(2) Enamels fish scale more readily when they are applied to both sides of the piece.

(3) Fish scaling is more likely to occur when the piece is fired in air than when the oxidation-control method of firing is used. Careful control of the amount of oxidation of the steel surface is necessary. Excessive iron oxide concentrations at the interface destroy the bubble structure of the enamel and promote fish scaling.

(4) Enamels fish scale more readily when the water-vapor content of the furnace is high than when it is low. Laboratory work shows that lowering the dew point of the furnace atmosphere definitely reduces the fish-scaling tendency, which verifies the statements of Zapffe and Sims and of Hoff and Klärding. These investigators state that the reaction of water and of the base metal is the chief source of the trouble-making hydrogen.

(5) Clear-firing clays make fish scaling worse, and clays which impart a good bubble structure tend to reduce fish scaling. There appears to be two reasons for these conclusions. The opaque clays have a lower content of chemically combined water and therefore less water available for the hydrogen-producing reaction. The opaque clays also impart a bubble structure to the enamel which must make the coating more permeable to the entrapped gas than would be obtained with a clear-firing clay. The bubble structure of the opaque-firing clays apparently is due to their organic material content.

(6) Cold-working of the metal surface reduces the tendency to fish scale. An explanation of this has already been given in the quotation from Zapffe and Faust,[5] who contend that cold-working opens the structure of the metal and permits effusion of the hydrogen during the drying operation.

(7) A gas is given off at the fish-scaling points which often continues to evolve for several hours. This gas proved by analysis to be almost pure hydrogen. Hydrogen then is the gas at the metal-enamel interface which causes the formation of fish scales.

This conclusion, together with the other results shown in this paper, is the basis for two general statements as follows: (*a*) these data substantiate the hydrogen theory as proposed by Zapffe and Sims; and (*b*) some practical applications of the hydrogen theory are also shown, for example, how the tendency to fish scale may be minimized or eliminated. The use of proper enameling clay and low water vapor in the furnace atmosphere, the use of only the rim and core portion of rimmed-type steel wherever practical and cold-working of the metal surface, and last (but certainly not the least), the use of the oxidation-control method of firing are practical things which may be done to minimize the occurrence of this defect.

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